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Fabrication and Microstructure of Metal-Metal Syntactic Foams

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Abstract

The objective of this paper is to report on the fabrication process and document the microstructure of metal-metal syntactic foams. The composite microstructure consists of thin-wall, hollow Fe-Cr stainless steel spheres cast in various metal matrices including aluminum alloys 6061, 7075, 413, magnesium alloy AZ31B, and unalloyed aluminum and magnesium. Hollow spheres were formed from slurries of fine particle size iron and chromium oxides via the coaxial nozzle process. The oxides were reduced and sintered in hydrogen to the corresponding Fe-Cr alloy. Spheres were sufficiently uniform to allow arrangement into random or periodic arrays. These arrays were infiltrated by an aspiration casting process, resulting in a structure less dense than the matrix metal.

1 Introduction

Economically produced metallic foams having low density and high strength are potentially useful in a wide variety of applications [1]. These materials show great promise as replacements for bearing and support structures, for impact and sound absorption, and where the advantages of both metallic character and low density are required. In many industries, especially aerospace and automotive, the need for weight and cost reduction without a compromise in mechanical properties has become a dominant driver [2].

Syntactic foams represent a class of composite materials consisting of hollow spheres embedded in a matrix. These foams exhibit low density, good energy absorption capability, and high compressive strengths [3-6]. Previous syntactic structures using either aluminum or magnesium alloys and containing oxide hollow spheres have demonstrated their potential use as energy absorbing materials [4]. Most currently available metal foams rely on the introduction of pore structure through high shear mixing or chemical decomposition [7]. The low viscosity and high surface tension of liquid metals result in a wide distribution of pore sizes and foams that exhibit inconsistent mechanical behavior. Infiltrating molten metal around a preform of uniform hollow spheres allows control of porosity by changing the size and packing arrangement of the spheres.

While much has been learned about structures of varied sphere-matrix material combinations, metal-metal syntactic foams have not been explored. The superior bulk properties of metals, notably strength and ductility, enhance the properties of foams constructed from these systems and can provide distinct advantages over alternative lightweight materials. This paper outlines the process and apparatus used to fabricate

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metal-metal syntactic foams, microstructural analysis of several metal-metal syntactic foams, preliminary methods to control microstructure, and areas for future research.

2 Experimental Procedure

Hollow spheres comprising the foam preform were made from slurries of fine particle size (1-10 μm diameter range, 6 μm average diameter) iron and chromium oxides via the coaxial nozzle process [2]. The sphere formation apparatus consists of a pair of coaxial nozzles. The inner jet sustains a flow of an inert gas while the annulus formed between the inner and outer jets carries slurry. The slurry is a high solids content, low viscosity combination of solvent, polymer binders, and dispersants. The slurry solvent evaporates, leaving behind a polymer bonded, hollow ceramic sphere. These oxide spheres were reduced in hydrogen to their constituent alloy and arranged into the desired preform array. The preform may consist of packed individual spheres or a rigid, diffusion bonded structure.

The aspiration casting apparatus, shown schematically in Fig. 1, consists of a fused silica casting tube, an intermediate vacuum chamber, and a rough vacuum pump. The casting tube contains the preform to be infiltrated. A cordierite honeycomb flow straightener was positioned below the preform to promote uniform infiltration during casting. Ceramic fiber insulation was packed above the preform into the remainder of the tube. The high surface area of the insulation provides shear resistance to flow following infiltration. This resistance slows the flow of metal enough to prevent significant penetration beyond the top of the preform. The intermediate vacuum chamber serves as a secondary safety measure and prevents damage to the rough vacuum pump from high temperature fluids.

During the casting process, matrix material is superheated in a crucible to approximately 100 °C above the liquidus temperature. The end of the casting tube is then lowered into the melt. When the vacuum pump is turned on, molten metal flows into the tube and infiltrates the preform as the tube is evacuated. Complete infiltration of the spheres is determined by visual inspection.

When Al was used to infiltrate the preform, the casting tube was water quenched after the vacuum pump was turned off. Quenching the casting minimizes the effects of reactions

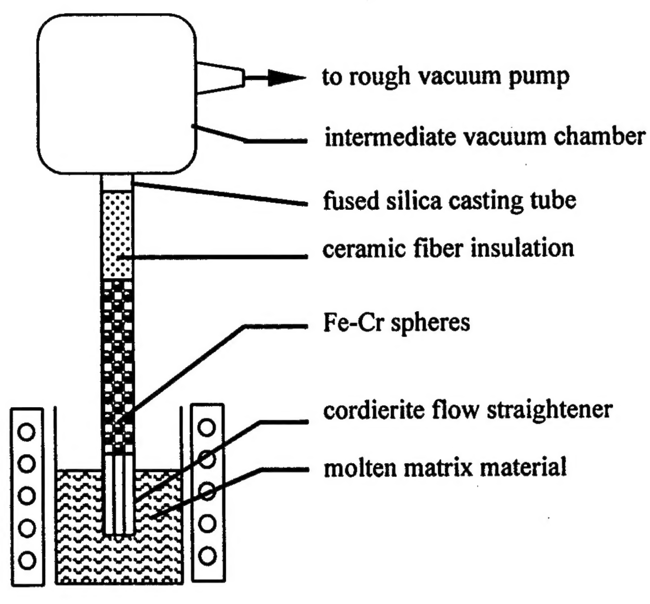

Fig 1: Schematic of aspiration casting apparatus.

between the preform and the matrix. During the quench, the volume change of the casting causes the surrounding glass tube to crack and fall away. When casting Mg, additional setup and process adjustments were required. The Mg melt was covered with a $MgCl_2$ flux and a flow of argon to minimize reaction with the atmosphere. After sphere infiltration, the casting tube was removed from the melt and placed in $MgCl_2$ to prevent reaction of the exposed metal. The glass casting tube cracked and fell away upon air cooling. Samples were sectioned and polished, and examined using a Reichart optical microscope, model MeF3a.

3 Results and Discussion

Several aluminum and magnesium alloys have been used as a matrix material. Aluminum alloys included 6061, 7075, 413, and unalloyed Al. Magnesium alloys included AZ31B and unalloyed Mg. Microstructural analysis revealed no porosity in any of the castings (i.e. complete infiltration), and complete wetting of the spheres by the matrix as shown in Figs. 2-5. Fig. 2 shows the microstructure of Fe-Cr hollow spheres cast in a 7075 Al matrix. Inset is an actual size photograph of the same foam. Spheres that are nonhermetic due to flaws or excessive porosity may be infiltrated, increasing density unnecessarily. By changing sphere formation parameters and heat treating schedules, hermeticity can be greatly improved.

In the aluminum matrix foams, there is an interaction between the sphere wall material and the matrix. The interaction results in a deleterious, faceted phase surrounding each sphere and extending into the matrix. Fig. 3 shows Fe-Cr spheres in a eutectic Al-Si (413) alloy. When magnesium was used as a matrix material, as shown in Figs. 4 and 5, there was no detectable interfacial reaction. Fig. 4 shows the microstructure of Fe-Cr hollow spheres cast in an unalloyed Mg matrix. Fig. 5 shows the microstructure of spheres cast in an AZ31B Mg alloy matrix. Inset is a photograph of the diffusion bonded preform to be infiltrated.

To minimize the interaction between the Fe-Cr spheres and the aluminum matrix, a passivation process was employed. Passivating the spheres increased the thickness of the surface Cr_2O_3 layer. Batches of spheres were held at 400 °C in air for ½, 1, 2, 4, and 8 hours and subsequently cast in unalloyed Al. None of the passivated batches of spheres were able to completely stop the reaction between preform and matrix, however using passivation in conjunction with rapid quenching greatly reduced the thickness of the reacted layer. Detailed results of these efforts will be reported in later work. Future work will also focus on the improvement of hollow sphere processing and an evaluation of mechanical properties.

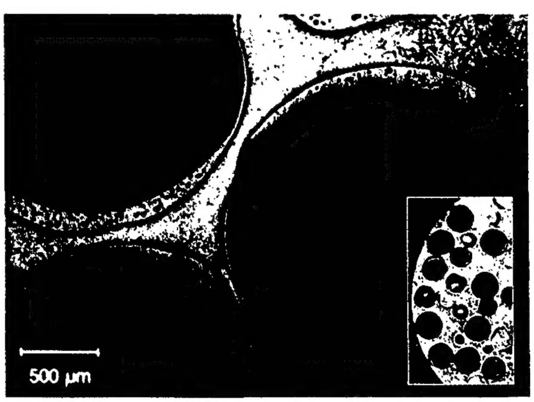

Fig 2: Fe-Cr hollow spheres in a 7075 Al alloy matrix. Inset: Bulk syntactic foam, spheres ~3 mm diameter.

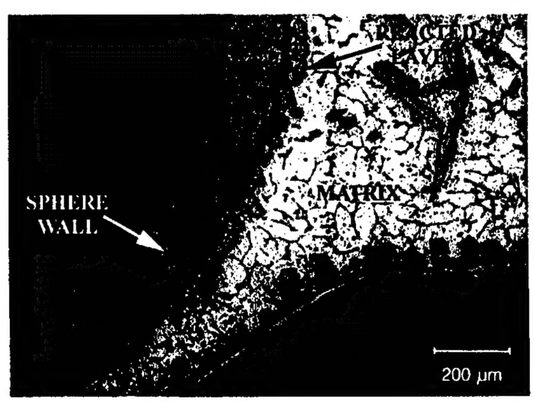

Fig 3: Fe-Cr hollow spheres in a 413 Al alloy matrix showing reacted layer at the sphere-matrix interface.

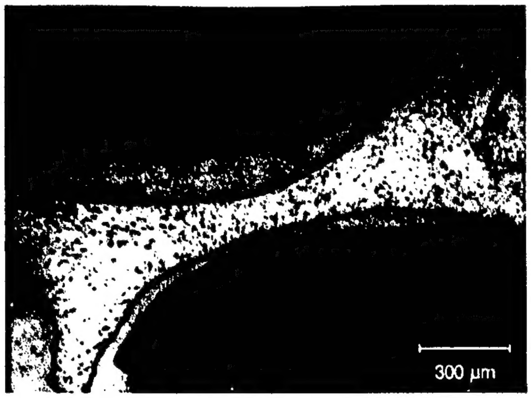

Fig 4: Fe-Cr hollow spheres in a Mg matrix.

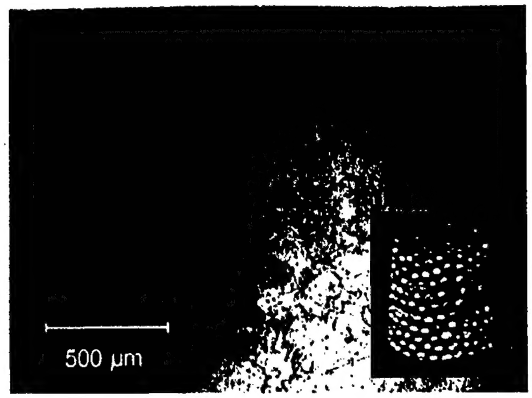

Fig 5: Fe-Cr hollow spheres in AZ31B Mg alloy matrix. Inset: Diffusion bonded preform, ~2 cm diameter.

4 Conclusions

Metal-metal syntactic foams were manufactured using an aspiration casting process. The composite foam consists of thin-wall, hollow metal spheres cast in various metal matrices including aluminum alloys 6061, 7075, 413, magnesium alloy AZ31B, and unalloyed aluminum and magnesium. In aluminum matrix foams, there is a reaction between the sphere wall material and the matrix. Using passivation in conjunction with rapid quenching greatly reduced the thickness of the reacted layer. Magnesium and its alloys show promise as a non-reacting matrix material.

References

[1] J. K. Cochran, "Ceramic hollow spheres and their applications", *Current Opinion in Solid State & Materials Science*, no. 3, 1998, pp. 474-479.

[2] K. M. Hurysz, J. L. Clark, A. R. Nagel, C. U. Hardwicke, K. J. Lee, J. K. Cochran, and T. H. Sanders, Jr., "Steel and titanium hollow sphere foams" in Porous and Cellular Materials for Structural Applications, MRS Proceedings vol. 521, edited by D. Schwartz, *et al.*, (MRS, Pittsburgh, PA, 1998), pp. 191-203.

[3] M. Hartmann, K. Reindel, and R. Singer, "Fabrication and properties of syntactic magnesium foams", in Porous and Cellular Materials for Structural Applications, MRS Proceedings vol. 521, ed. by D. Schwartz, *et al.*, (MRS, Pittsburgh, PA, 1998), pp. 211-216.

[4] S. A. Rickles, J. K. Cochran, and T. H. Sanders, Jr., "The production and compressive characteristics of a low density syntactic metal/ceramic foam", in Proceedings of the 13th annual Conference on Composites and Advanced Materials, edited by R. E. Banks, (American Ceramics Society, Westerville, OH, 1989) pp. 1472-1484.

[5] P. Bunn and J. T. Mottram, "Manufacture and compression properties of syntactic foams", *Composites*, Vol. 24, No. 7, 1993, pp. 565-571.

[6] M. T. Kiser, M. He, B. Wu, and F. W. Zok, "Compressive deformation of hollow microsphere reinforced metal matrix composites" in Spheres and Microspheres: Synthesis and Applications, MRS Proceedings vol. 372, edited by D. L. Wilcox, *et al.*, (MRS, Pittsburgh, PA, 1995), pp. 269-275.

[7] J. Banhart and P. Weigand, "Powder metallurgical process for the production of metallic foams", in Proceedings of the Fraunhofer USA Metal Foam Symposium, edited by J. Banhart and H. Eifert, (MIT-Verlag, Bremen, 1997), pp. 13-22.